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Investigating the controllable factors influencing the weight loss of grinding ball using SEM/EDX analysis and RSM model

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ABSTRACT

This study was aimed to investigate the effects of individual variables and their interactions on weight loss of grinding ball using the SEM/EDX analysis and RSM model. The results SEM/EDX analysis indicated that corrosion mechanism type for steel balls is pitting. In addition, abrasive corrosion was observed on high carbon chromium steel ball surface. It was also found that steel balls were formed from corrosion phases of Fe, O, S and Si. The results of RSM model showed that the linear effects of all factors and the quadratic effects of solid concentration and charge weight of balls were significant parameters on wear rate. Also, it was observed no interactions between factors. Furthermore, results indicated that degree of influence of factors on the wear rate was in the order of ball type > solid percentage > pH > solid percentage^2 > charge weight^2 > grinding time > rotation speed of mill > charge weight > throughout.

1. Introduction

Ball mills are the most common and versatile type of grinding mills. They are remarkable in that they can operate over a very wide range of conditions and geometries. Ball mills may be used for primary, secondary, tertiary and regrind applications. In a mineral processing plant, they are often used for a secondary grinding duty, which directly links with downstream recovery processes such as flotation [21,22]. Mild steel or stainless steel balls are generally used as grinding media in ball mills for processing of sulphide ores to achieve the required liberation size. Grinding circuit operators have long been aware of the significant impact of grinding media consumption on the cost of grinding. It is estimated that comminution includes 30–50% of typical mining operating costs, and of these, liner wear and media consumption account for roughly 50% of the cost [1,19]. Therefore consumption of grinding media forms a significant part of the operating cost in mineral processing industry. According to Ref. [11] grinding medium wear can constitute up to 40–45% of the total operation cost in comminution process.

Total media wear in ball mills is a product of three recognized wear mechanisms including abrasion, corrosion and impact [20]. Also, the contributions to total media wear of each of these wear mechanisms has not been well established [24]. Abrasion and impact wear is metal loss due to mechanical force on the grinding media. Erosion wear results from the friction between grinding media and particles. Corrosive wear is defined as metal loss due to chemical and/or electrochemical reactions of grinding media with the solution and/or other electrochemical conductive particles [6,16].

There are many different factors which could influence on the mass losses of grinding media. These factors can be summarized under five headings [1,2,4–7,9,10,13,15–18,23,24]:

(1) The grinding media: where composition and metallurgical properties of the grinding media, grinding media size, grinding media size distribution, hardness, shape, charge weight and media selection methodology are the most important parameters.
(2) The ore: where particles sizes, hardness (abrasiveness), work index, density, shape and ore mineralogy and lithology are the most important parameters.
(3) The mill: where size, speed and discharge type are the most important parameters.
(4) The grinding environment: where pH, percent solid, viscosity, Eh, gas purging (air, oxygen and nitrogen), temperature, rheological properties, water chemistry and galvanic interaction between grinding media and mineral in wet grinding are the most important parameters.

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Table 1
Characterization of Sarcheshmeh ore representative sample.

Chemical compositions	SiO_2	Al_2O_3	CaO	MgO	K_2O	MnO	Na_2O	P_2O_5	TiO_2	Fe_2O_3	SO_3	Cu	Mo	LOI
Weight, %	59.47	14.35	0.96	2.39	3.72	0.06	0.74	0.18	0.54	6.2	7.62	0.74	0.032	2.17

Table 2
Chemical compositions of the grinding media.

Ball type	Chemical compositions (weight, %)							
	C	Si	S	P	Mn	Cr	Mo	Cu
High carbon chromium steel (HS)	2.28	0.698	0.049	0	1	13.25	0.177	0.044
Low alloy steel (LS)	0.249	0.173	0.024	0.018	0.586	0.019	0.002	0.012

(5) The grinding circuit: where throughout (input feed to ball mill), circulating load and grinding time are the most important parameters.

Based on these observations and the importance of wear in milling costs, this paper was aimed to investigate some of controllable factors affecting the mass loss of grinding ball. These factors were pH, solid percentage (pulp density), throughout of ball mill, rotation speed of mill, charge weight of balls and grinding time.

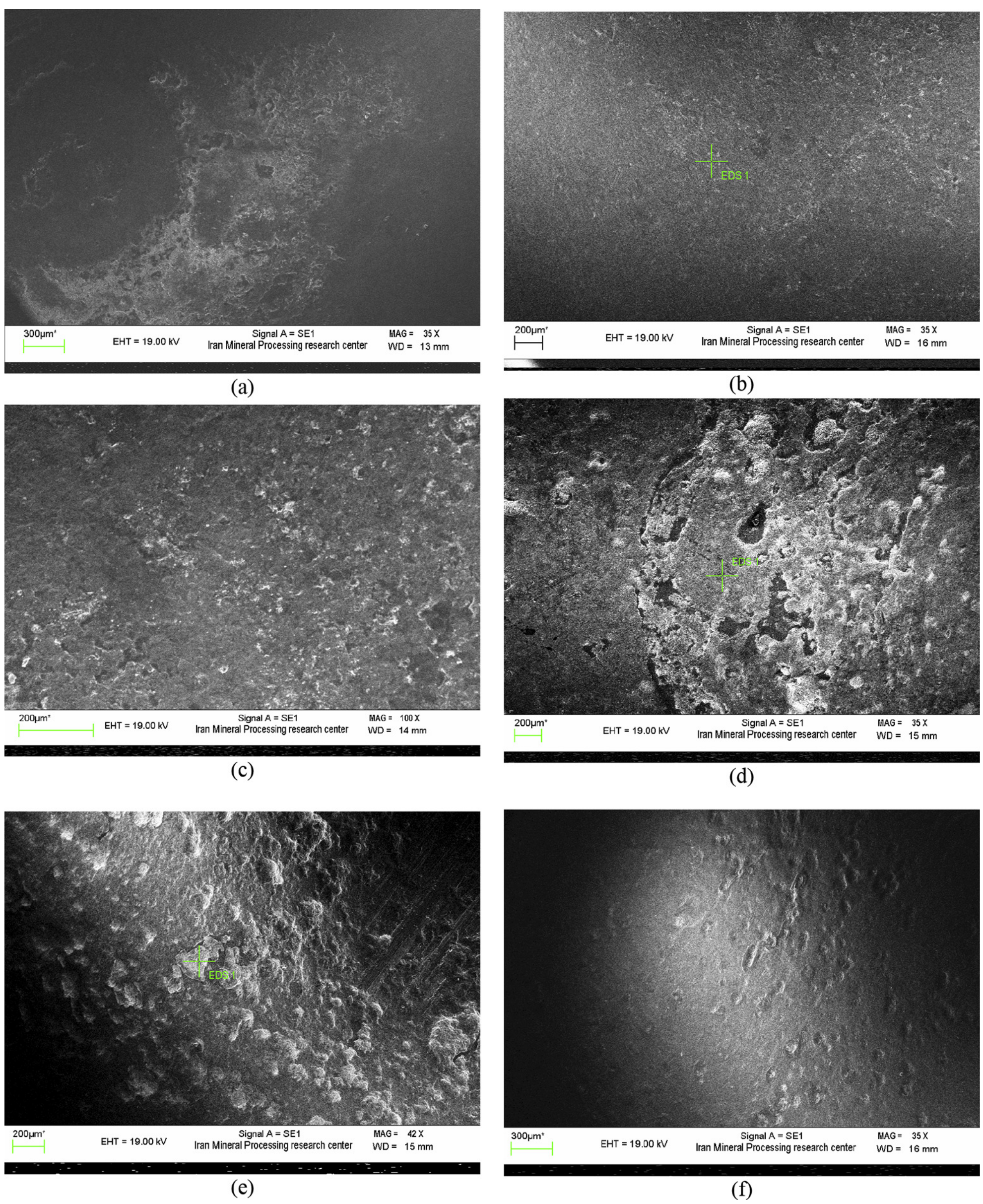

Fig. 1. SEM images of steel balls under grinding different conditions; pH: 7, percent solid: 35, grinding time: 60 min, mill speed: 75 rpm and ball type: low alloy steel (a); grinding time: 90 min (b); grinding time: 90 min and mill speed: 85 rpm (c); grinding time: 300 min (d); grinding time: 300 min and ball type: high carbon chromium steel ball (e); pH: 10, percent solid: 55 and ball type: high carbon chromium steel ball (f).

This research was carried out on the Sarcheshmeh copper sulfide ore. Sarcheshmeh ore is a major porphyry copper deposit, which is located in Kerman Province in the southeastern part of Iran. Sarcheshmeh Copper Mine is the largest copper producer in Iran, and one of the major producers in the world market [3].

2. Methodology

2.1. The SEM/EDX

Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDX) is a powerful technique applied in microimaging of a variety surfaces. This technique can be used in exploring the surface structure to determine particle size and texture on that surface. The SEM/EDX allows the study of both morphology and composition of biological and physical materials. In addition, detailed maps of elemental distribution can be produced from multi-phase materials or complex, bio-active materials. Characterization of fine particulate matter in terms of size, shape, and distribution as well as statistical analyses of these parameters, may be performed [23]. In this research, mechanism type and products of corrosion has been studied using scanning electron microscopy (Zeiss, LEO 435vp, United Kingdom).

2.2. The RSM model

Response surface methodology (RSM) is empirical statistical tools that are being used for modeling and analyzing problems where the response is influenced by several independent variables and the aim is to get an optimum response [12]. This methodology is widely used in chemical engineering and applied sciences to optimize process variables. As described RSM usually contain three steps: (a) design and experiments, (b) response surface modeling through regression, (c) optimization [14]. In this study, this technique was employed to assess the main and interactive effects of the parameters and also to model and minimize the wear of the steel balls.

3. Experimental

3.1. Materials

The obtained samples from the ball mills input of the Sarcheshmeh copper concentrator plant were crushed in a jaw crusher (Fritsch 01.703). The size fraction of $-2000 + 250$ μm was collected for experiments. Samples were chemically analyzed which their chemical analysis presented in Table 1.

Steel balls used in Sarcheshmeh concentrator plant were employed as grinding media, which their chemical compositions presented in Table 2.

3.2. Grinding

Samples (365–720 g) were ground under different experimental conditions in a laboratory ball mill (drum mill) with 6–18 kg ball (mixing of 0.5, 0.75 and 1 inch ball in diameter with equal weights) at grinding time, 10–15 min and rotation speed, 70–80 rpm such as 70% of particles were less than 75 μm in diameter. The ball mill used was included a stainless steel pipe with 21 cm of diameter and length of 30 cm with a wall thickness of 0.7 cm.

In determining the ball mass losses through total wear, 15 steel balls were handpicked and marked and then before and after each grinding experiment were weighted to calculate the ball losses. Then, the wear rate in mils penetration per year (mpy) was calculated according to [8] using the following formula.

$$\text{mpy} = \frac{534 \times W}{\rho \times A \times t} \tag{1}$$

where W is weight loss in milligrams, ρ is density in grams per cubic centimeter, A is area in square inches, and t is time in hours.

4. Results and discussion

Since the aim of study was to evaluate the effects of selected parameters on the mass loss of steel balls within grinding, scanning electron microscopy with energy dispersive X-ray detection (SEM–EDX) and the statistical design of experiments were employed to investigate the mass loss products of steel balls and to evaluate the effects of operating parameters on the wear rate, respectively. These operating parameters were pH, solid percentage, mill throughout, charge weight of balls, rotation speed of mill, grinding time and steel ball type.

The SEM/EDX instrument is a powerful and flexible tool for solving a wide range of product and processing problems for a diverse range of metals and materials. SEM–EDX was used to obtain information about the elemental distributions in the

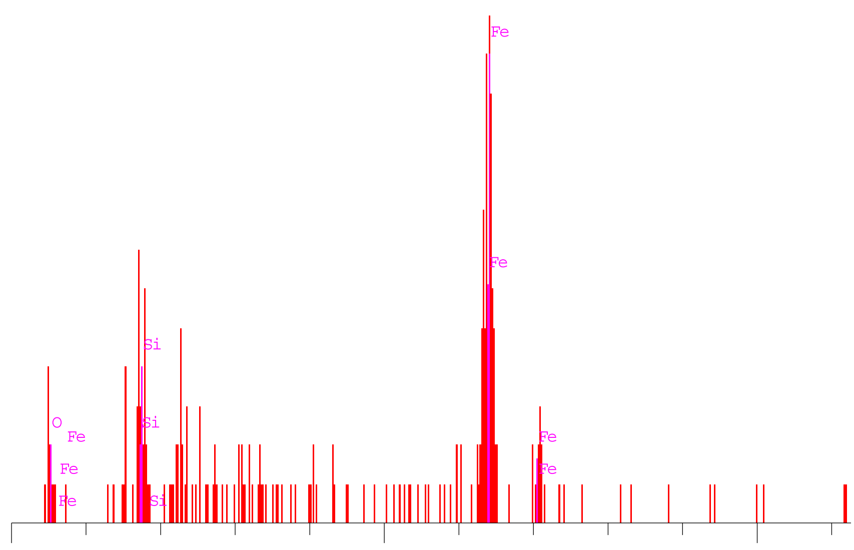

Fig. 2. EDX analysis of low alloy steel balls under grinding conditions; pH 7, percent solid 35, grinding time 90 min and mill speed 75 rpm (EDX analysis for Fig. 1b).

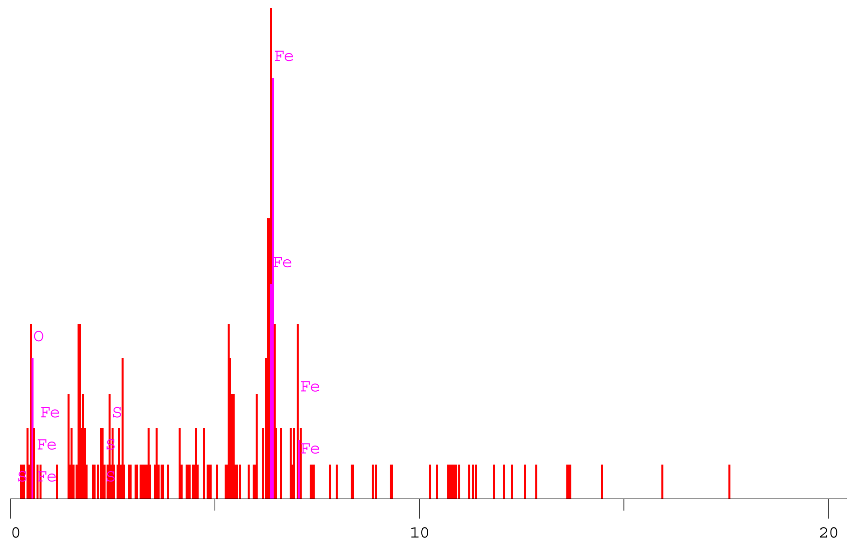

Fig. 3. EDX analysis of low alloy steel balls under grinding conditions; pH 7, percent solid 35, grinding time 300 min and mill speed 75 rpm (EDX analysis for Fig. 1d).

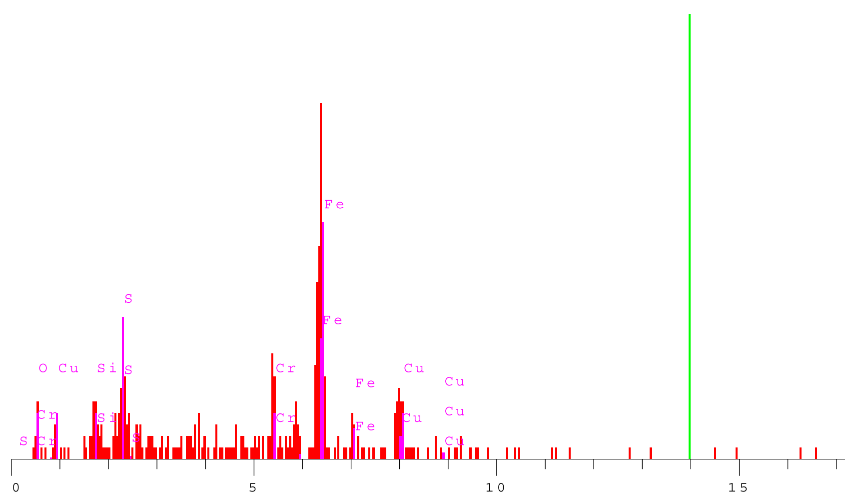

Fig. 4. EDX analysis of high carbon chromium steel balls under grinding conditions; pH 7, percent solid 35, grinding time 90 min and mill speed 75 rpm (EDX analysis for Fig. 1e).

corrosion layers. To obtain the samples for SEM–EDX analysis, the grinding time was changed from 1 to 5 h. The microstructures of the corroded surfaces of the steel balls obtained at grinding different conditions. The results of SEM–EDX analysis are presented in Figs. 1–4. These figures indicate a change in the grinding conditions strongly influenced the mass loss of balls, type and products of corrosion. The analysis via SEM–EDX has allowed to check the degree of corrosion suffered by the various test-pieces exposed to different environments and to confirm that the highest categories of corrosion correspond to rougher layers of corrosion products. As can be observed in Fig. 1, the gouges and scratches on the balls surface indicate that the mass loss of steel balls is due to corrosion and abrasion mechanism. The darker spots might be due to the formation of an oxide or dark metal composition while the brighter spots might be due to bright metal composition (for example chromium). Also, Fig. 1 clearly shows that there are a lot of deep and shallow pits on the surface. According to SEM/EDX observations can be concluded that pitting corrosion is the main corrosion mechanism. Pitting corrosion is caused by localized attack in an otherwise resistant surface. In addition, abrasive effects are seen on surface of high carbon chromium steel balls (Fig. 1e). It is observed that an increase in pH and solid percentage and change of ball type from low alloy steel ball to high carbon chromium steel ball, deep and value of pits on surface of steel balls reduce (Fig. 1e). This behavior indicates the reduction of corrosion on the surface. The analyses of EDX (Figs. 2–4) indicate that corrosion products of high carbon chromium steel balls are phases of Fe, O, S, Cr and Si while corrosion products of low alloy steel balls are Fe, O, S and Si.

Table 3
Selected parameters and their actual (and coded) levels.

Factor	Symbol	Type of factor	Low Actual	High Actual	Low Coded	High Coded
pH	A	Numeric	8	10	−1	1
Solid content (%)	B	Numeric	30	50	−1	1
Throughout (g)	C	Numeric	360	720	−1	1
Charge weight (kg)	D	Numeric	8	12	−1	1
Speed (rpm)	E	Numeric	70	80	−1	1
Grinding time (min)	F	Numeric	10	15	−1	1
Ball type	G	Categoric	High steel	Low alloy steel		

Table 4
The conducted experiments conditions (D-optimal design of experiment) and calculated values of wear rate of steel balls.

Run	pH	Solid %	Throughout (g)	Charge weight (kg)	Speed (rpm)	Grinding time (min)	Ball type	Wear rate (mpy)
1	8	35	720	12	70	10	LS	462.11
2	10	45	360	12	80	10	HS	213.72
3	10	35	360	12	70	10	HS	251.52
4	10	45	360	12	70	15	HS	218.33
5	9	50	540	10	75	12.5	LS	317.66
6	9	40	500	11	75	12.5	HS	236.79
7	8	35	720	12	70	10	HS	286.11
8	10	45	360	8	70	10	LS	292.91
9	9	30	540	10	75	12.5	HS	360.23
10	8	45	360	12	80	15	HS	337.61
11	10	35	720	8	70	10	HS	224.48
12	8	45	360	12	80	15	LS	515.51
13	10	35	720	8	80	15	LS	408.26
14	9	40	600	10	70	12.5	LS	311.95
15	10	35	720	12	70	15	LS	447.67
16	9	40	540	10	75	11	HS	198.03
17	8	35	360	8	70	15	LS	478.46
18	8	35	720	8	80	10	HS	299.59
19	10	45	720	12	70	10	LS	335.18
20	8	45	720	8	80	15	LS	469.95
21	10	35	720	12	80	10	HS	257.68
22	8	45	360	8	80	10	HS	261.47
23	9	40	540	10	75	12.5	LS	387.04
24	10	45	720	8	70	15	HS	199.21
25	9	40	540	10	75	12.5	LS	391.58
26	10	45	360	8	80	15	HS	232.91
27	8	35	360	8	80	10	LS	488.36
28	8	45	720	8	80	10	LS	403.43
29	9	40	540	10	75	12.5	HS	256.45
30	8	45	720	8	80	15	HS	288.25
31	8	45	720	8	70	10	LS	366.3
32	8	35	360	12	70	15	HS	365.17
33	10	45	720	8	80	10	LS	347.4
34	8	45	720	12	70	15	HS	260.78
35	8	35	360	8	80	15	LS	555.53
36	10	35	720	12	80	10	LS	442.73
37	8	35	360	8	70	10	HS	293.64
38	8	45	720	8	70	10	HS	226.05
39	10	35	360	12	80	15	LS	475.28
40	8	35	720	8	80	15	HS	357.47
41	8	35	360	8	70	10	LS	463.27
42	9	40	540	10	75	12.5	HS	236.84
43	10	45	720	12	70	10	HS	182.79
44	8	35	720	8	70	15	LS	484.98
45	10	45	720	12	80	15	HS	241.03
46	8	45	360	8	70	15	LS	435.97
47	10	35	360	8	80	10	HS	230.18
48	10	40	700	10	75	12.5	LS	303.22
49	8	40	600	10	75	12.5	LS	439.87
50	10	35	720	8	80	15	HS	259.74

After studying the corrosion and wear of balls by SEM/EDX, operating parameters influencing the wear rate of steel balls were quantitatively evaluated using experimental design technique, response surface methodology (RSM). Tables 3 and 4 show selected parameters levels and wear rate calculated under experimental different conditions based on response surface model by a D-optimal design of experiment, respectively.

Analysis of experimental data with fitting a response surface model was done which the results are presented in Table 5 and Figs. 5 and 6.

The results in Table 5 and Figs. 5 and 6 reveal that the linear effects of all of factors and the quadratic effects of solid percentage and balls charge weight are statistically significant in the mass loss of balls. It is also observed no interactions between factors. In addition, It is found that the influence of individual variables and their quadratic effects on the wear rate of steel balls is in the order of ball type > solid percentage > pH > solid percentage^2 > charge weight^2 > grinding time > rotation speed of mill > charge weight > throughout. In addition, the regression equation for balls wear rate (CR) as coded variables is:

$$\begin{aligned} \mathrm{CR} = {} & 300.92 - 39.8 \times A - 66.73 \times B - 7.84 \times C + 15.06 \times D \\ & + 17.99 \times E + 21.34 \times F + 78.55 \times G + 38.16 \times B^2 \\ & + 38.03 \times D^2 \end{aligned} \tag{2}$$

Moreover, the relationship between independent and dependent variables was graphically represented by 3D response surface. The 3D response surface plot is a graphical representation of the regression equation. It is plotted to understand the interaction of the variables and locate the optimal level of each variable for minimum response. Each response surface plotted represents the different combinations of two test variables at one time while maintaining the other variable at the zero level (center level). Figs. 5 and 6 indicate influence of two variables on wear rate of steel

Table 5
Analysis of variance (ANOVA) for quadratic model for wear rate of steel balls.

Source	Sum of squares	df	Mean square	*F* value	*p*-Value Prob > F	
Model	4.76E+05	9	52,901.81	132.82	<0.0001	Significant
A-pH	57,645.76	1	57,645.76	144.73	<0.0001	
B-solid content	51,911.83	1	51,911.83	130.33	<0.0001	
C-throughout	2368.53	1	2368.53	5.95	0.0193	
D-charge weight	7671.1	1	7671.1	19.26	<0.0001	
E-speed	12,240.44	1	12,240.44	30.73	<0.0001	
F-grinding time	17,234.57	1	17,234.57	43.27	<0.0001	
G-ball type	2.96E+05	1	2.96E+05	743.23	<0.0001	
B^2	2387.91	1	2387.91	6	0.0188	
D^2	11,460.21	1	11,460.21	28.77	<0.0001	
Residual	15,932.12	40	398.3			
Lack of fit	15,729.53	38	413.94	4.09	0.2158	Not significant
Pure error	202.58	2	101.29			
Cor total	4.92E+05	49				
Std. dev.	19.96	*R*-squared	0.9676			
Mean	336.01	Adj *R*-squared	0.9603			
C.V. %	5.94	Pred *R*-squared	0.93			
PRESS	34,422.11	Adeq precision	41.193			

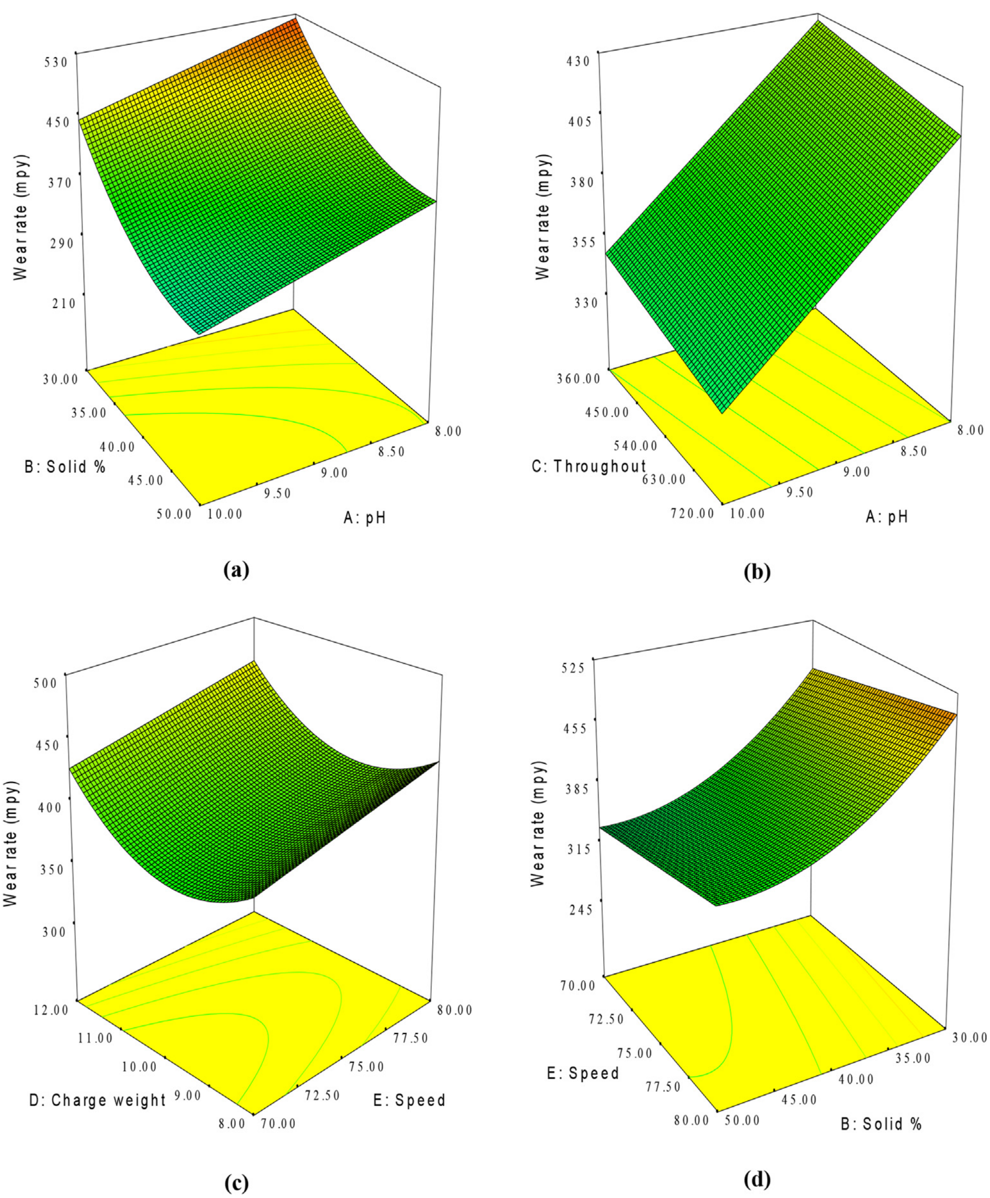

Fig. 5. Response surface plots showing the effect of two variables on wear rate of low alloy steel balls (other variables are held at center level): (a) pH and solid percentage; (b) pH and throughout; (c) charge weight and rotation speed of mill; (d) solid percentage and rotation speed of mill.

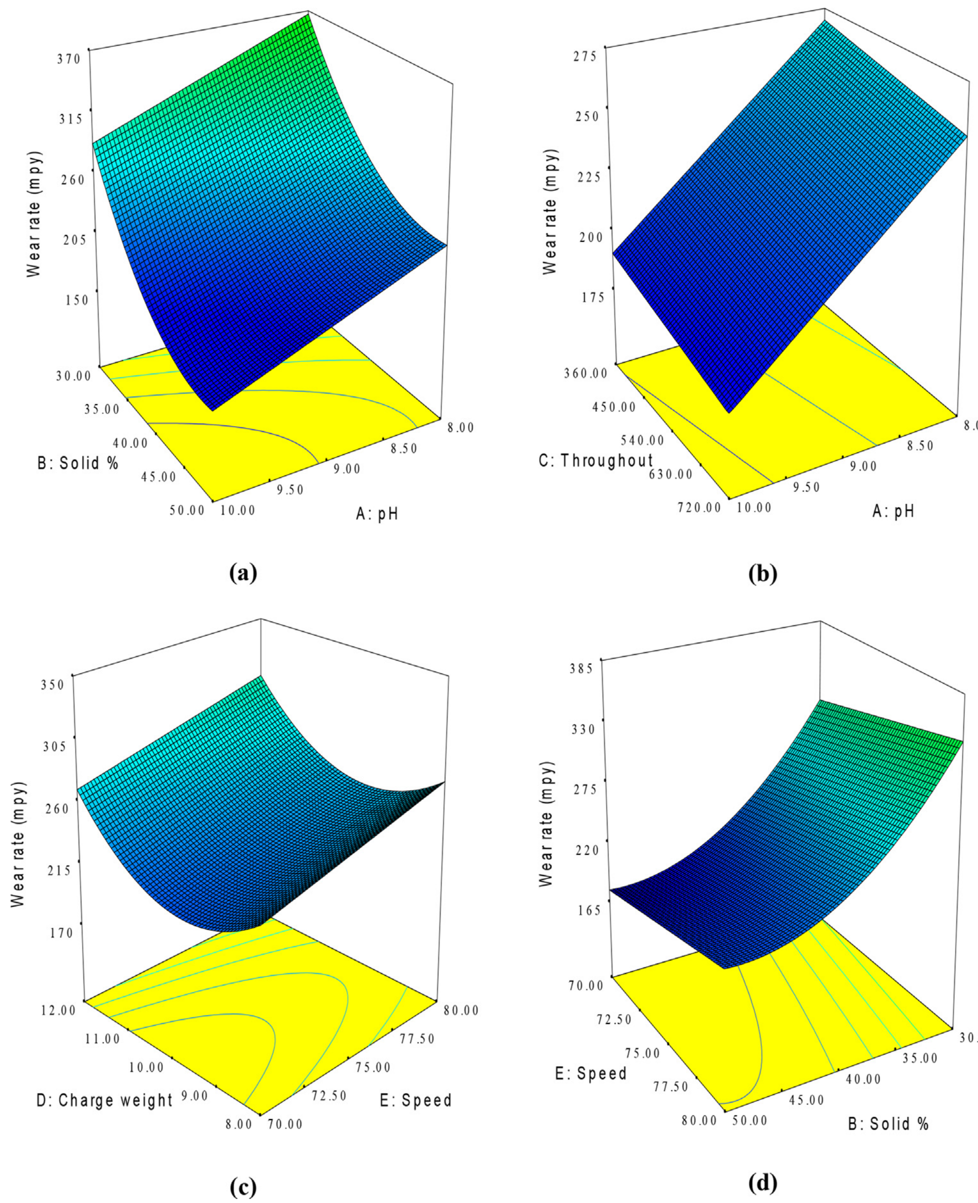

Fig. 6. Response surface plots showing the effect of two variables on wear rate of high carbon chromium steel balls (other variables are held at center level): (a) pH and solid percentage; (b) pH and throughout; (c) charge weight and rotation speed of mill; (d) solid percentage and rotation speed of mill.

balls when other variables are held at center level. The following observations obtained from Figs. 5 and 6:

Increase of pH and solid content improved the mass loss of steel balls. This is possibility attributed to the fact that increasing pH caused to formation of the passive ferric oxide film. Ferrous oxide is an insulator that prevents electron transfer, hindering corrosion and the weight loss of metal (or ball). On the other hand, increasing feed solids percentage increased the pulp viscosity which affected the abrasion rate. At higher pulp viscosity, the motion of the balls and the copper sulphide samples were reduced significantly. If the pulp viscosity was too high, the balls and the sulphide samples would be stuck on the mill shell, significantly reducing the abrasion rate and at lower feed solids percentage; more oxygen will be dissolved in the pulp. Dissolved oxygen in aqueous solution had profound influence on the corrosion of metal. It was also seen that pH and solid content had effects of linear and quadratic on wear rate, respectively.

The wear rate increased with increasing the rotation speed of mill in range investigated. According to Ref. [23]; this may be caused by the fact that the balls are lifted higher at high rotation speed and the balls have stronger impacts on the mill shell upon falling on to the coupon, which increases the abrasive wear. It was mentioned, if the rotation speed was close to or higher than the critical speed, the wear rate was reduced significantly because the media were held against the shell by centrifugal force and no abrasive wear happened at this speed range.

It was observed that wear rate reduced with increasing the charge weight of balls to a certain value and then with further increasing enhanced.

It was obvious that wear rate decreased with increasing the mill throughout. However this factor had the little influence on wear rate. This behavior is attributed to the fact that increasing mill throughout reduced the motion space of the balls, reducing the contact chance between the balls and the mill shell.

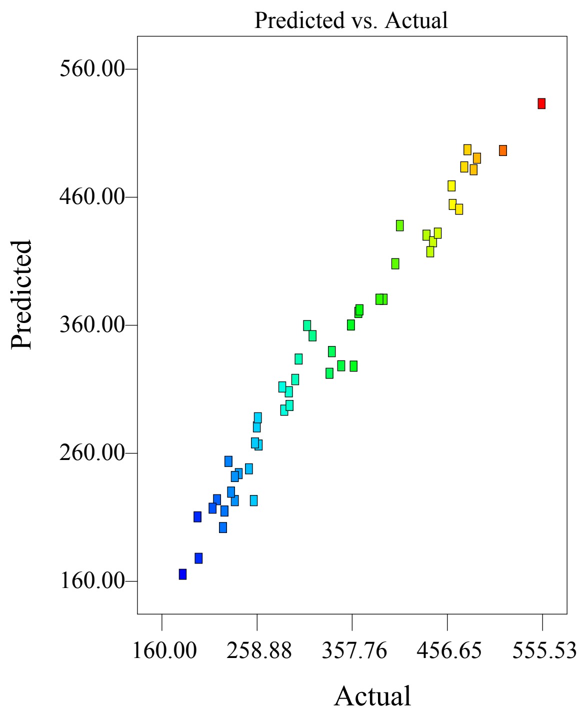

Fig. 7. The predicted values of wear rate using regression model versus actual values.

Wear rate were strongly affected by ball type. The high carbon chromium steel balls resulted in the fewer weight loss of balls.

Also, the predicted values of wear rate were obtained using the model equation and were plotted versus experimental data which are shown in Fig. 7. According to Fig. 7 and Table 5, predicted values are in very good agreement with the measured values (R^2 value of 0.9676, adjusted R^2 value of 0.9603 and predicted R^2 value of 0.93).

5. Conclusions

In this study, wear rate of steel balls within the grinding of Sarcheshmeh copper sulphide ore was qualitatively investigated using SEM/EDX analysis. Also, factors affecting the mass loss of balls including pH and solid percentage of grinding environment, throughout of mill, charge weight of balls, mill speed, grinding time and type of steel balls were evaluated by using RMS model. The major conclusions based on this paper can be summarized as follows:

1) The SEM images of high carbon chromium steel balls were compared with low alloy steel balls. The results displayed that the corrosion of low alloy steel was much more serious than that of high carbon chromium steel under the same conditions. The SEM/EDX analysis showed that the weight loss of balls is due to pitting corrosion and abrasion. The corrosion products formed from phases of Fe, O, S, Si and Cr. The low alloy steel balls that depend on a passive film for corrosion resistance were especially susceptible to pitting by local breakdown of the film at isolated sites. In addition, the SEM images indicated that there are fewer scratches and pits on low alloy steel ball surface.
2) Increase of pulp pH and solid content strongly reduced wear rate of steel balls. It was observed that wear rate enhanced with reducing the throughout and increasing the grinding time and mill speed. In addition, the ball charge weight enhanced up to certain value, the wear rate decreased and thereafter increased.
3) It was found that influence of factors affecting the wear rate of steel balls was in the order of ball type > solid percentage > pH > solid percentage^2 > charge weight^2 > grinding time > rotation speed of mill > throughout.
4) A mathematical model was also suggested for relationship between operating important factors and wear rate of balls.

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